

## X-RAY DIFFRACTION OF SOIL SAMPLES FROM OKWUTA IBEKU UMUAHIA, NIGERIA

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### ABSTRACT

Soil samples in Okwuta Ibeku Umuahia were characterized for their mineralogical potentials. X-ray diffraction was used in the mineralogical characterization. Result obtained was analyzed using Bragg-Wolf equation and International Centre for diffraction data software. Bragg's equation was used to find the d-value corresponding to Bragg's angles. The d-values which were obtained from samples were compared with standard values. The d-spacing for the most intense peaks in the x-ray diffraction pattern are 4.27013, 3.35360 and 1.82025Å. The result showed that the sample contained  $\alpha$ -quartz (SiO<sub>2</sub>) an hexagonal crystal system with Space group P3221, a (Å) : 4.9209, b (Å):4.9209, c (Å): 5.4091, alpha (°): 90.0000 beta (°):90.0000, gamma (°):120.0000, volume of cell (10<sup>6</sup> pm<sup>3</sup>):113.43 , Z: 3.00 ,RIR: 2.99

**KEYWORDS:** Soil; quartz; x-ray diffraction; d-spacing; minerals; Umuahia

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### INTRODUCTION

A mineral is defined as a naturally occurring homogeneous solid with a definite (but not generally fixed) chemical composition and an ordered atomic arrangement, and is usually formed by inorganic processes (Karathanasis and Hajek, 1982). Both chemical composition and crystal structure (ordered atomic arrangement) are important parts of this definition; neither alone is sufficient to explain the properties of minerals. Minerals make up about 50% of the volume of most soils. They provide physical support for plants and create the water and air-filled pores that make plant growth possible (Anthony *et al.*, 1995). Most of the elements in the crust and in soil occur in minerals. Identifying minerals is the first step in mineral analysis. Sometimes, one single analytical method is sufficient to accomplish the job, and sometimes, several analytical techniques have to be employed. Among commonly-used methods, X-ray diffraction (XRD) has been proven to be indispensable (Deng *et al.*, 2009; Bailey, 1991). The primary goals of mineralogy analyses are: to identify mineral species, to quantify mineral composition, and to reveal mineral structures that determine the chemical reactivity of the minerals (Janet *et al.*, 2001). Many techniques can be used for mineralogy analysis, such as petrographic microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), electron diffraction, neutron diffraction, Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, nuclear magnetic resonance (NMR), electron paramagnetic resonance (EPR), Mossbauer spectroscopy, chemical analysis, computational modeling, and many more (Farmer, 1974; Harris and White, 2008; White, 2008; Francoise *et al.*, 2008; Warren, 2008; April and Richard, 2008). Each technique has its own merits and drawbacks. One technique may not be equally suitable for different samples or for different fractions of one particular sample. X-ray powder diffraction is the best available technique for the identification and quantification of all minerals present in clay-rich rocks (claystones and mudstones). Accurate quantitative mineral analysis is important in petrological studies, engineering, and industrial applications of rocks that contain clay minerals (Harris and White, 2008). Harris and White stressed x-ray diffraction (XRD) is the technique most heavily relied in soil mineralogical analysis. X-ray diffraction is a technique that provides detailed



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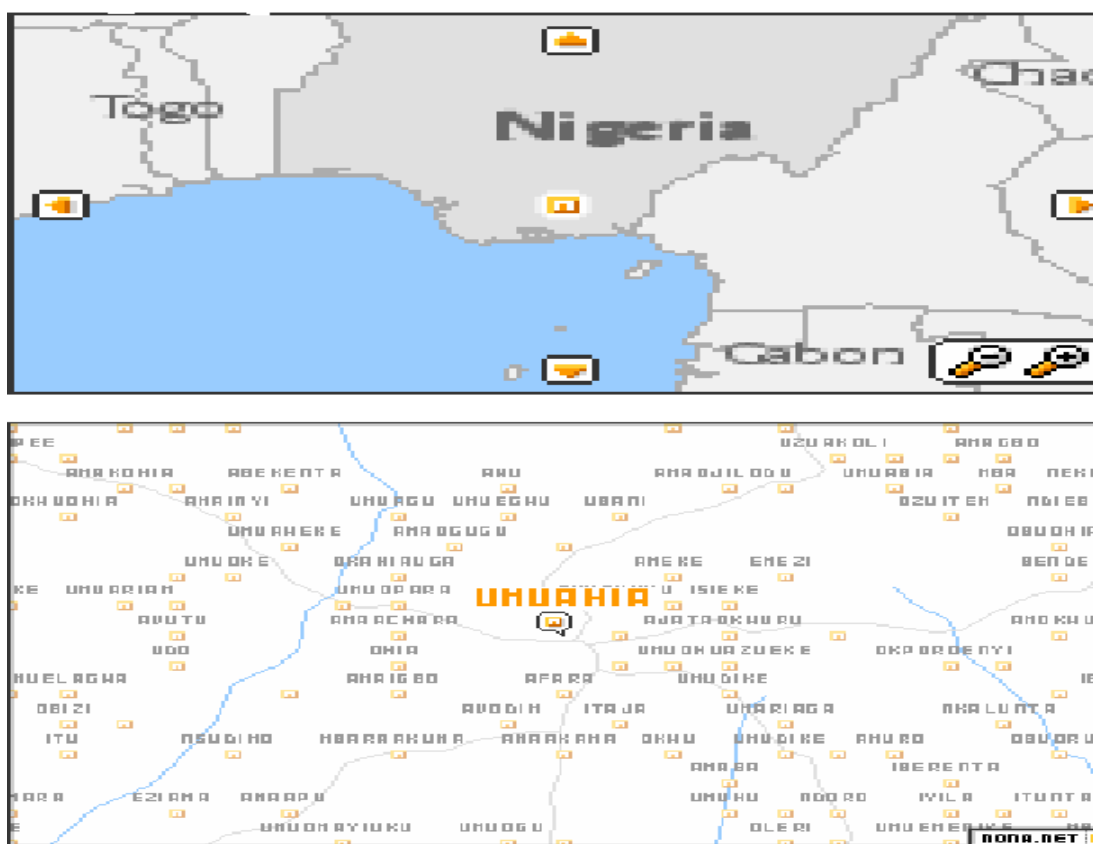
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information about the atomic structure of crystalline substances (White, 2008). It is a powerful tool in the identification of minerals in rocks and soils. The bulk of the clay fraction of many soils is crystalline, but clay particles are too small for optical crystallographic methods to be applied. Therefore, XRD has long been a mainstay in the identification of clay-sized minerals in soils. Mineral identification is relatively simple and unambiguous if modern software and good mineral databases are available, accurate quantitative analysis of clays remains a formidable challenge (Victor 2001). We hereby present the x-ray diffraction of soil sample B from Okwuta Ibeku, Umuahia, Nigeria.

## MATERIALS AND METHODS

### *Description of the Study Area*

The study area is located within Latitude (lat): 5°32'0"N and Longitude (lon): 7°29'0"E



### *Sample Collection*

Soil sample B was collected from Okwuta Ibeku Umuahia at a depth of 10-15 cm. The sample was packed in a small plastic bag and labeled. The collected sample B was strained to remove the fibrous and undesired materials with the help of muslin cloth and dried by heating to remove humidity because humidity affects the results very badly and then ground to make them homogeneous before x-ray powder diffraction (XRPD) analysis. Particle size determination was made by the hydrometer method (Bouyoucos, 1962). Bulk density was determined by Core method (Grossman and Reinsch, 2002). Colour was compared with standards. pH was determined using Ciba Corning pH meter.

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Qualitative phase analysis by Hanawalt's method (Harris and White, 2008).

X-ray diffraction patterns were made with a monochromatic X-ray Diffractometer system XPERT-PRO with beta filter CuK $\alpha$  radiation wavelength of 1.5406Å and automated silt. A set of 2 $\theta$  angle ranging from 10°- 90° was used; this was done at Sheda Science and Technology Complex (SHESTCO), Abuja Nigeria. Qualitative phase analysis was used for the study of crystal structure and unknown phases of soil.

## RESULTS AND DISCUSSION

The physical characterization of soil sample B is presented in Table 1. The x-ray diffractogram of sample B is reported in Figure 1 while its peak list is reported in Table 2. The reference pattern from ICDD using POWD-12++ have been presented in Figure 2.

Table 1: Physical characterization of soil Sample B

|                                  |                        |
|----------------------------------|------------------------|
| Particle Size distribution (PSD) | Clay 56%               |
|                                  | Silt 28%               |
|                                  | Sand 16%               |
| Colour                           | Light Brown            |
| pH                               | 5.9                    |
| Bulk Density                     | 0.82 g/cm <sup>3</sup> |

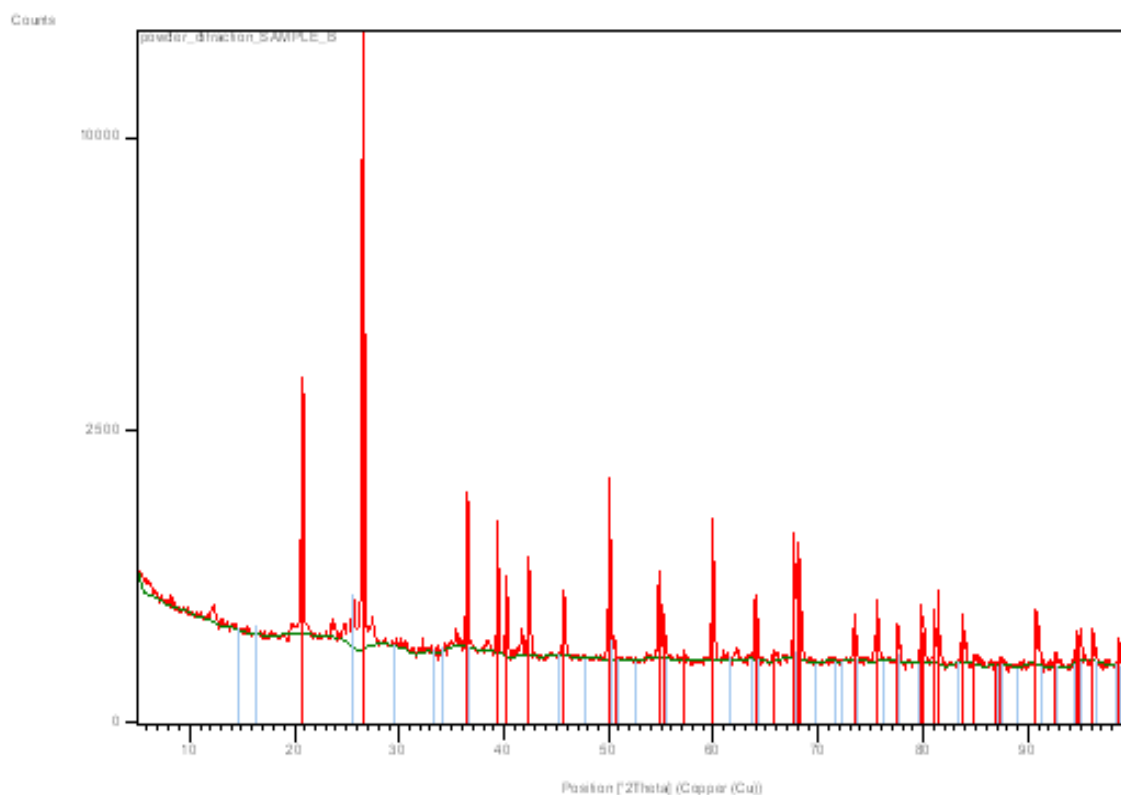


Figure 1: X-Ray Diffractogram for Sample B



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| Table 2: Peak List for Sample B |             |             |              |             |
|---------------------------------|-------------|-------------|--------------|-------------|
| Pos.[°2Th.]                     | Height[cts] | FWHM[°2Th.] | d-spacing[Å] | Rel.Int.[%] |
| 12.2940                         | 109.31      | 0.5274      | 7.19965      | 0.75        |
| 20.8027                         | 3359.33     | 0.1978      | 4.27013      | 23.10       |
| 23.7343                         | 64.66       | 0.3956      | 3.74891      | 0.44        |
| 25.5952                         | 190.41      | 0.3956      | 3.48041      | 1.31        |
| 26.5804                         | 14541.73    | 0.1978      | 3.35360      | 100.00      |
| 36.5110                         | 1352.22     | 0.1978      | 2.46106      | 9.30        |
| 39.4362                         | 1007.55     | 0.1978      | 2.28498      | 6.93        |
| 40.2597                         | 529.94      | 0.1978      | 2.24013      | 3.64        |
| 42.4173                         | 608.67      | 0.1978      | 2.13104      | 4.19        |
| 45.7641                         | 332.77      | 0.1978      | 1.98268      | 2.29        |
| 50.1158                         | 1134.82     | 0.1978      | 1.82025      | 7.80        |
| 54.8429                         | 519.42      | 0.1978      | 1.67401      | 3.57        |
| 59.9331                         | 1042.54     | 0.2637      | 1.54344      | 7.17        |
| 64.0305                         | 304.89      | 0.2637      | 1.45420      | 2.10        |
| 67.6834                         | 930.35      | 0.1978      | 1.38434      | 6.40        |
| 68.1745                         | 469.00      | 0.2637      | 1.37556      | 3.23        |
| 73.3952                         | 172.93      | 0.2637      | 1.29007      | 1.19        |
| 75.5818                         | 331.66      | 0.3296      | 1.25809      | 2.28        |
| 77.5713                         | 161.70      | 0.1978      | 1.23073      | 1.11        |
| 79.8321                         | 269.80      | 0.3296      | 1.20146      | 1.86        |
| 81.3993                         | 192.94      | 0.5274      | 1.18225      | 1.33        |
| 83.7375                         | 196.20      | 0.1978      | 1.15509      | 1.35        |
| 90.7469                         | 290.46      | 0.1319      | 1.08323      | 2.00        |
| 94.5869                         | 99.59       | 0.1978      | 1.04913      | 0.68        |
| 96.2628                         | 80.38       | 0.3956      | 1.03526      | 0.55        |
| 98.6701                         | 107.07      | 0.2412      | 1.01551      | 0.74        |

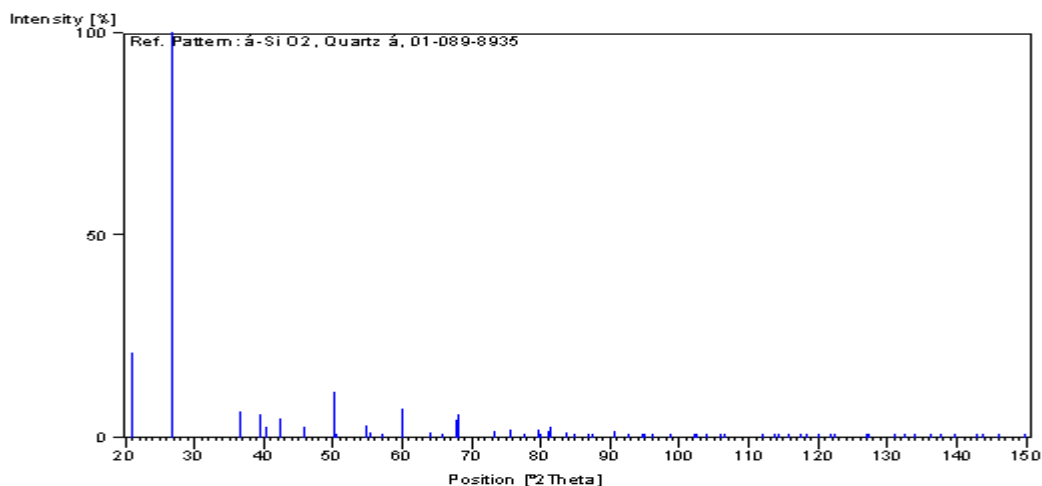


Figure 2: The reference stick pattern from ICDD using POWD-12++



*Name and formula*

|                    |                                              |
|--------------------|----------------------------------------------|
| Reference code:    | 01-089-8935                                  |
| Common name:       | $\alpha$ -SiO <sub>2</sub> , Quartz $\alpha$ |
| PDF index name:    | Silicon Oxide                                |
| Empirical formula: | O <sub>2</sub> Si                            |
| Chemical formula:  | SiO <sub>2</sub>                             |

*Crystallographic parameters*

|                                                    |           |
|----------------------------------------------------|-----------|
| Crystal system:                                    | Hexagonal |
| Space group:                                       | P3221     |
| Space group number:                                | 154       |
| a (Å):                                             | 4.9209    |
| b (Å):                                             | 4.9209    |
| c (Å):                                             | 5.4091    |
| Alpha (°):                                         | 90.0000   |
| Beta (°):                                          | 90.0000   |
| Gamma (°):                                         | 120.0000  |
| Volume of cell (10 <sup>6</sup> pm <sup>3</sup> ): | 113.43    |
| Z:                                                 | 3.00      |
| RIR:                                               | 2.99      |

In XRPD pattern, there were two parameters (Bragg's angle and integrated intensities). Bragg's equation was used to find the d-value corresponding to Bragg's angles. The d-values which were obtained from samples were compared with standard values. This was done by employing International centre for diffraction data software. The d-spacing for the most intense peaks in the x-ray diffraction pattern of sample B are 4.27013 , 3.35360 and 1.82025 Å compare well the d-spacing in the reference stick pattern for  $\alpha$ -quartz 4.26160, 3.34750 and 1.82000 Å . Other d-spacing values of the diffractogram compare well with the stick pattern. The existence of  $\alpha$ -quartz mineral with a hexagonal crystal system and Space group P3221 was confirmed.

Quartz is a piezoelectric material. It has long been used for controlling frequency of radio transmitters, and it has been essential component in telecommunication equipment. Quartz has great economic importance. Many varieties are gemstone. Large amount of quartz sand (also known as silica sand) are used in the manufacture of glass and ceramics. Crushed quartz is used as an abrasive in sand paper. Silica glass (also called fused glass) is used in optics to transmit ultraviolet light. The identification of  $\alpha$ -quartz in Okwuta, Ibeku Umuahia is of enormous advantage because quartz can now be mined locally in Nigeria, This will reduce the importation of quartz into Nigeria.

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